

# Execution of Comprehensive Performance Test Using Particulate, HCl and Metals Continuous Emissions Monitoring Systems

Paper #26

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## ABSTRACT

Evonik Degussa Corporation, Tippecanoe Laboratories successfully demonstrated compliance with the HWC MACT Comprehensive Performance Test (CPT) requirements utilizing the continuous emission monitors for particulate matter (PM), hydrochloric acid (HCl), and metals as the compliance analyses for stack emissions. It is the authors' belief that this is the first CPT conducted in the United States utilizing this approach.

This paper will briefly review the history of the CEMs development on the solid-liquid incinerator, provide an overview of the CPT plan and objectives, discuss the performance of the CEMs prior to and during the CPT, and present the performance test results. This performance test demonstration illustrates that the use of CEMs for performance test demonstration is an efficient, effective, and viable approach for compliance demonstration on hazardous waste incinerators.

## INTRODUCTION

Evonik Degussa Corporation (Evonik) operates a Hazardous Waste Incinerator that burns solids and liquid organic and aqueous waste at their newly acquired facility, Tippecanoe Laboratories (Tippe), in Lafayette, IN. The site was acquired by Evonik in 2010 from Eli Lilly and Company (Lilly) and has continued to manufacture pharmaceutical and animal health compounds for Lilly.

Selected incinerator operating parameters limits and emissions limits are specified by the U. S. EPA Hazardous Waste Combustor Maximum Achievable Control Technology (HWC MACT) rule in order to minimize emissions of Hazardous Air Pollutants (HAPs). Since the early 1990s when the MACT rules were initially being considered, EPA has often expressed a desire to use Continuous Emissions Measurement Systems (CEMS) technology as a means to directly demonstrate compliance by measuring emissions directly, rather than use operating parameters to infer compliance. Several years ago Tippe embarked on a program to evaluate and apply available CEMS technologies for particulate matter and hydrogen chloride, and to assist in developing a novel CEMS technology for metals.

Evonik successfully completed a Comprehensive Performance Test (CPT) in 2010 utilizing CEMs for HCl, particulate and the HWC/MACT metals in lieu of standard EPA test methods for those pollutants. It is the authors' belief that this is the first CPT conducted in the United States utilizing this approach.

## **HISTORY AND BACKGROUND**

A previous Lilly affiliate facility in Ireland employed particulate matter (PM) CEMS and infrared multi-component CEMS under a different regulatory structure for a several years as indicators of compliance. EPA included the requirement for the application of PM CEMS to hazardous waste incinerators in the proposed HWC MACT rule in 1996. Tippe, along with a number of industry peers, demonstrated the limitations of these technologies on certain types of hazardous waste incineration systems. EPA subsequently deferred the requirement to implement PM CEMS under the HWC MACT. While demonstrating the viability of the PM CEMS, Tippe joined forces with Cooper Environmental Services to develop practical application of X-ray fluorescence as a multi-metals CEMS technology use on a solid-liquid waste incinerator.

Tippe has been involved with the testing, evaluation and development of CEMs beginning with installations on the liquids incinerators in the 1990s and subsequently on the solids and liquids incinerators in the early 2000s.

This development of the use of these CEMS technologies met a couple key Tippe concerns. First, using CEMS data for compliance reduced or eliminated the need for sampling and analysis of individual containers to develop waste characterization. This addressed the concerns Tippe had regarding the costs and potential exposure required by typical sampling and analytical programs. Due to the nature of high-potency pharmaceutical wastes, reducing or eliminating the typical sampling and analysis activities provided significant safety, operational, and economic benefits.

Second, Tippe was also interested in the benefits from operational flexibility that could be realized by removing many of the prescribed HWC MACT operating parameters limits on the incinerator's air pollution control system.

Tippe installed continuous monitoring of the combustion (stack) gas emissions on the solid and liquid waste incinerator designated T149 utilizing three CEMS:

- An EcoChem MC3 or equivalent for CO, O<sub>2</sub>, and HCl
- A Sigrist CTNR or equivalent for particulate emissions
- A Cooper Environmental Services XACT or equivalent for metals.

Since the required use of CEMs for parameters other than CO and O<sub>2</sub> was removed from the HWC MACT requirements, the use of CEMs for PM, HCl and multi-metals required the submission and approval of an alternative monitoring petition. The requirements for petitioning the EPA for alternative monitoring for compliance is found in 40 CFR Part 63.8 (f), the General Provisions of the National Emission Standards for Hazardous Air Pollutants (NESHAP) for Source Categories. Paragraph (f)(4)(ii) states:

“The application must contain a description of the proposed alternative monitoring system which addresses the four elements contained in the definition

of monitoring in §63.2 and a performance evaluation test plan, if required, as specified in paragraph (e)(3) of this section. In addition, the application must include information justifying the owner or operator's request for an alternative monitoring method, such as the technical or economic infeasibility, or the impracticality, of the affected source using the required method.”

Tippe initiated the process for the alternative monitoring petition (AMP) in early 2003. The AMP was approved by the EPA in January 2006. Implementation of the AMP then required a major modification to the facility's Title V permit.

## CPT OBJECTIVES

To demonstrate compliance with the HWC MACT performance standards and emission limits, Tippe proposed a single, low-temperature, combustion chamber test condition, treating solid and liquid wastes with enhanced ash, metals and chloride contents. This single test condition demonstrated the ability of the combustion system to comply with the applicable performance standards at the worst-case conditions of minimum combustion temperatures and maximum combustion air flow, and maximum waste, ash, and chloride feed rates.

Since T149 has not been modified in a way which would affect its ability to meet the DRE standard since DRE testing was conducted in September 2005, Tippe did not conduct a DRE test during the testing described here. This is in accordance with the provisions of 40 CFR 63.1206(b)(7)(i)(A), the HWC MACT Replacement Standard requires that compliance with the destruction and removal efficiency (DRE) standard must only be documented once, provided that the source is not modified after the DRE test in a way that could affect the ability of the source to achieve the DRE standard.

Under the provisions of the AMP approved in a letter dated January 27, 2006, the use of a particulate matter CEMS, a multi-metals CEMS, and an HCl CEMS to demonstrate compliance with the particulate matter, metals, and HCl/Cl<sub>2</sub> HWC MACT emission standards was approved and Tippe used that provision to demonstrate compliance for this CPT test.

The performance standards established that are applicable for the T149 incinerator are summarized in Table 1.

Table 1. HWC MACT Replacement Rule Performance Standards

Performance Standard	HWC MACT 40 CFR 63 Subpart EEE Replacement Standard-existing sources	
	Citation	Standard
Organic DRE	40 CFR 63.1219(c)(1)	99.99%
CO Emissions	40 CFR 63.1219(a)(5)(i)	100 ppm <sub>dv</sub> <sup>1</sup>
HC Emissions	40 CFR 63.1219(a)(5)(i)	10 ppm <sub>dv</sub> <sup>1</sup> , as propane
Particulate Emissions	40 CFR 63.1219(a)(7)	0.013 gr/dscf <sup>1</sup>
HCl/Cl <sub>2</sub> Emissions	40 CFR 63.1219(a)(6)	32 ppm <sub>dv</sub> HCl/Cl <sub>2</sub> combined as Cl <sup>-</sup> equivalents <sup>1</sup>
Metals Emissions	40 CFR 63.1219(a)(3)	Cd & Pb combined: 230 µg/dscm <sup>1</sup>

Performance Standard	HWC MACT 40 CFR 63 Subpart EEE Replacement Standard-existing sources	
	40 CFR 63.1219(a)(4)	As, Be & Cr combined: 92 µg/dscm <sup>1</sup>
	40 CFR 63.1219(a)(2)	Hg: 130 µg/dscm <sup>1</sup>
Dioxin/furan emissions	40 CFR 63.1219(a)(1)(ii)	0.40 ng/dscm TEQ <sup>1</sup>

1. CORRECTED TO 7% OXYGEN.

## CONTINUOUS MONITORING SYSTEMS PERFORMANCE EVALUATION TEST

As a part of the CPT, a performance evaluation of the CEMS was conducted in accordance with the CMS PET Plan. The evaluation showed that the Evonik's CMS was operating in compliance with the HWC MACT requirements [40 CFR 63.1209(a)-(b)] as:

The CEMS monitoring CO and O<sub>2</sub> stack gas concentrations met the appropriate performance specifications promulgated by the EPA.

The CEMS monitoring metals, HCl, and particulate met the appropriate performance specifications and requirements in the approved AMP using the annual auditing process.

Table 2. CEMS Audit Summary

Monitoring System	Audit Description	Audit Date	Result
CO CEMS	Relative Accuracy Test Audit (RATA)	8/24/2010	Pass
CO CEMS	Calibration Drift	8/18-24/2010	Pass
CO CEMS	Calibration Error	8/24/2010	Pass
CO CEMS	Response Time	8/24/2010	Pass
O <sub>2</sub> CEMS	Relative Accuracy Test Audit (RATA)	8/24/2010	Pass
O <sub>2</sub> CEMS	Calibration Drift	8/18-24/2010	Pass
O <sub>2</sub> CEMS	Calibration Error	8/24/2010	Pass
O <sub>2</sub> CEMS	Response Time	8/24/2010	Pass
PM CEMS	Response Correlation Audit (RCA)	8/11-12/2010	New Calibration Curve
PM CEMS	Absolute Correlation Audit (ACA)	8/12/2010	Pass
HCL CEMS	Accuracy (Dynamic Spiking)	8/24/2010	Pass
HCL CEMS	Seven Day Drift	8/22/2010	Pass
Metals CEMS	Total System Flow (Sample Volume)	8/4/2010	Pass

Metals CEMS	X-Ray Fluorescence Calibration Audit	8/9/2010	Pass
Metals CEMS	Accuracy (Dynamic Spiking)	8/5-6/2010	Pass

## HCl CEMS Audit Methodology

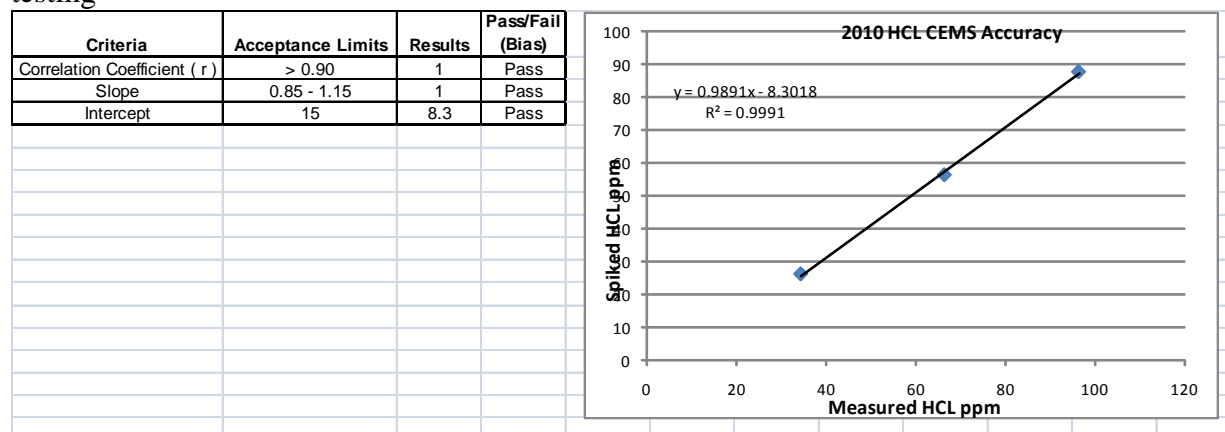
The accuracy and precision of the HCl CEMS is determined by dynamically spiking a known concentration of HCl reference gas into the sample system and subsequently measuring the reference spike with the HCl CEMS.

Daily, prior to testing, the zero and upscale drift is checked and recorded. The HCl CEMS must pass the daily calibration requirements prior to any testing.

This testing was conducted as part of the CMS performance evaluation test.

Figure 1 provides an overview of the regression analysis for the HCl dynamic spiking indicating the resulting performance for the HCl CEMS.

Figure 1. Regression analysis of the HCl dynamic spiking accuracy testing



## PM CEMS Audit Methodology

The objective of the annual relative calibration audit is to verify that the current calibration correlation is within statistical requirements for predicting total particulate mass based upon previous calibration to a particulate mass Reference Method (Method 5).

The RCA is performed by collecting a minimum of 12 valid (meets quality assurance requirements) particulate mass Reference Method samples (Method 5) over the expected operating range of the control device. The response of the PM CEMS is recorded during each Method 5 test and the data averaged to provide a PM CEMS response value (PLA) which correlates to a Method 5 data point (mg/scm).

Dual train Reference Method 5 sampling trains are used during the testing ensure the quality of the Reference Method particulate mass data.

Once all data is collected, both the Reference Method 5 and PM CEMS data are screened, according the Performance Specification 11 and Procedure 2 guidance, and only valid data is used for the calibration correlation check.

Using the current calibration correlation as the baseline, the Method 5 data is plotted against the respective PM CEMS averaged response. A minimum of 75% of the RCA data must fall within the current tolerance intervals of the existing calibration correlation.

If the new data does not meet this requirement, Performance Specification 11 provides guidance on how to establish a new or updated calibration correlation.

#### **PM CEMS Operating Range**

The operating range of the PM CEMS is 0 – 1 PLA. This range will be used for operation of the PM CEMS.

#### **PM CEMS Daily Linearity Check**

Daily, prior to any RCA testing, the linearity of the Sigrist photometer is checked. The Sigrist must pass applicable quality assurance requirements, daily, and prior to the relative calibration audit testing.

#### **Reference Method 5 Data Quality Assurance**

Reference Method 5 data collected during the RCA is analyzed to ensure it meets applicable quality control requirements. This is completed by performing relative standard deviation for each Method 5 test pair (dual train) along with linear regression analysis. In addition, a standardized residual test along with a bias check of the data may be used to ensure the Reference Method paired data sets are statistically sound for use in the relative calibration audit test. Reference Method data which does not meet the quality assurance guidance may be discarded, as long as a minimum of 12 Reference Method data points remain.

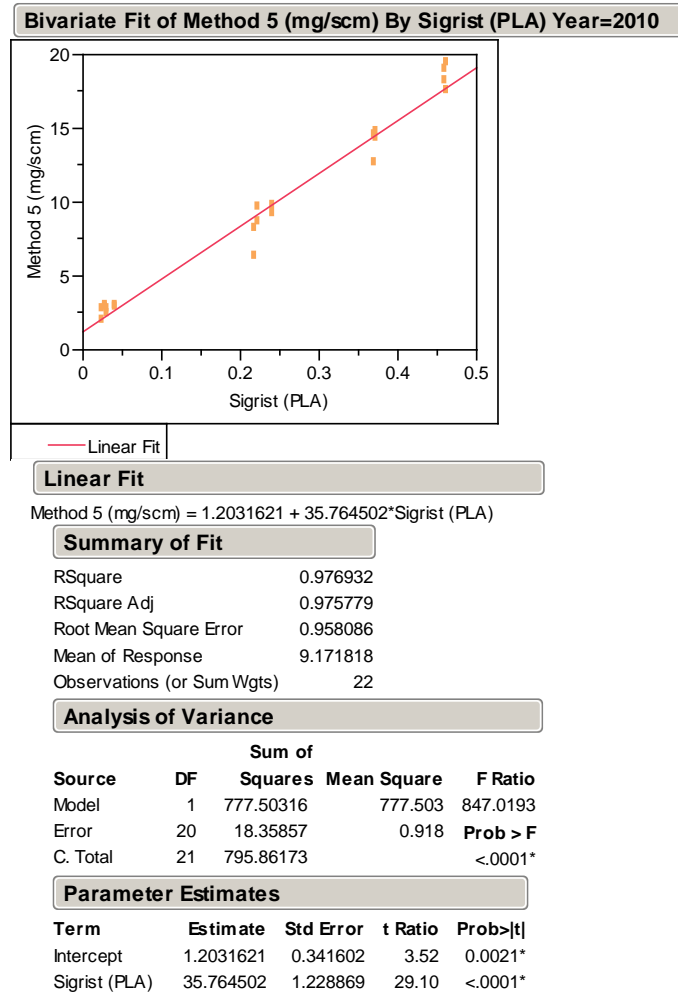


Figure 2. Calibration Correlation between the Sigrist PM CEMs and Method 5 train

### Particulate Emission Range of Data during the Relative Calibration Audit

The range of the total particulate emissions during the RCA should represent expected particulate emission levels during normal operation of the incinerator.

Per the T149 Alternative Monitoring Petition, Evonik will strive to provide a range of particulate emissions in which a minimum of 20% of the data falls into each of three ranges (1) 0-50% (2) 25-75% (3) 50-100%, with 100% being the highest Sigrist output observed during the testing. In addition, the data set of particulate mass emissions should fall within the data range represented by the current calibration correlation. The total particulate emission data collected, based upon the mg/scm concentrations, fell within three distinct ranges

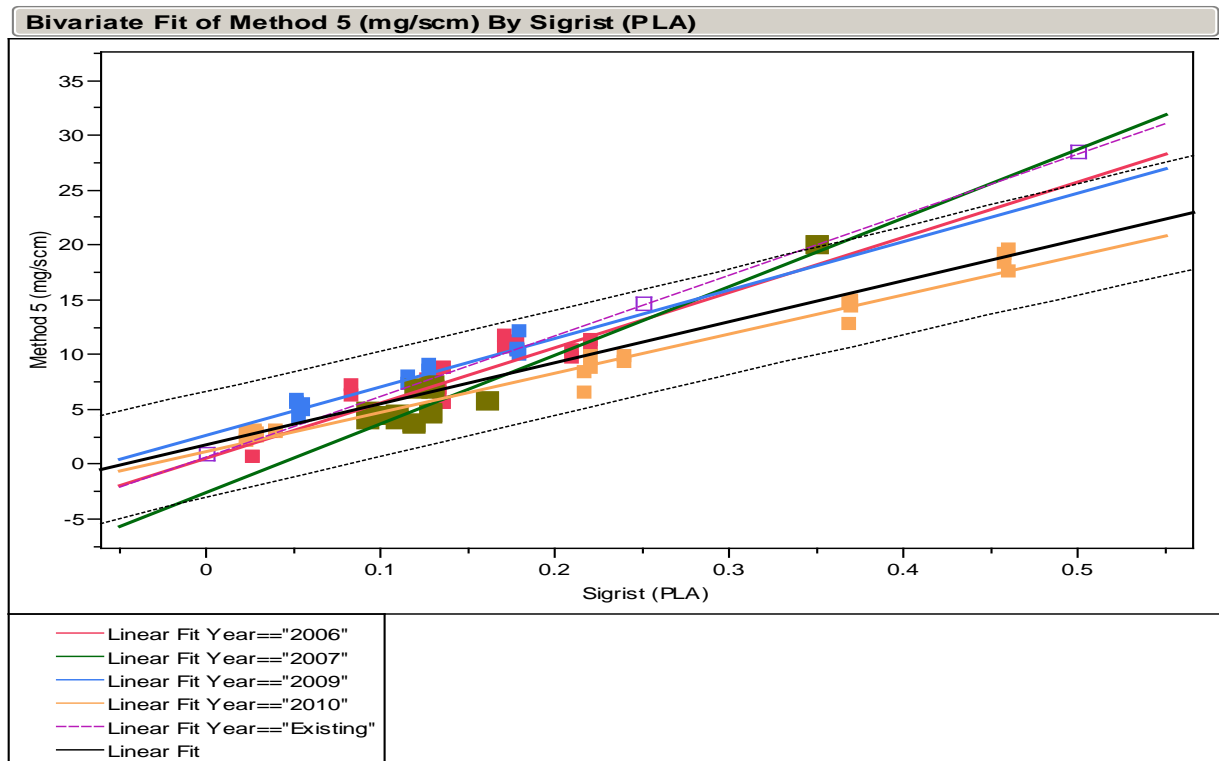


Figure 3. Performance Test Fit of Data for Sigrist PM CEMs

### Xact Multi-Metal CEMs Audit Methodology

The annual audit requirements for the Xact multi-metal CEMS are to perform a sample volume audit, a thin film standard audit, and an accuracy test. The accuracy test, per the T149 Alternative Monitoring Petition, is performed using dynamic spiking of lead, cadmium, arsenic, chromium, and mercury. Linear regression is used to assess the accuracy and precision of the Xact CEMS. The Xact CEMS met each of the audit requirements.

The accuracy and precision of the Xact CEMS is demonstrated by quantitatively spiking each of the regulated metals (arsenic, chromium, cadmium, lead, mercury) at a minimum of three levels. The metals are contained in a stock nitric acid solution and quantitatively spiked into the sample system, directly in back of the sample probe, using a quantitative aerosol generator. The concentration for each spiking trial is controlled by altering the mass loss rate of the aerosol generator and/or changing the concentration of the stock metals solution.

The range of metal concentrations tested encompasses the regulatory limits for each metal and are within the linear range of the Xact CEMS.



For each test level, a minimum of nine sample points are recorded. The output of the Xact multi-metals CEMS, for each MACT metal, is plotted against the reference metal value from the dynamic spiking. Regression analysis is performed and the correlation coefficient, slope, and intercept, for each MACT metal recorded.

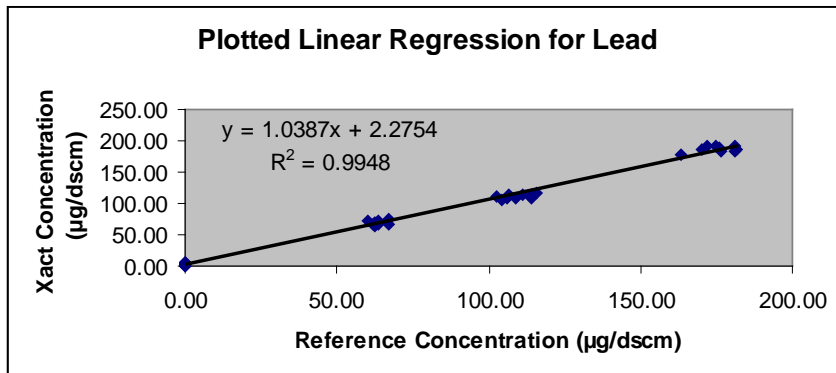


Figure 4. Plot of Linear Regression for Lead from Xact CEMs

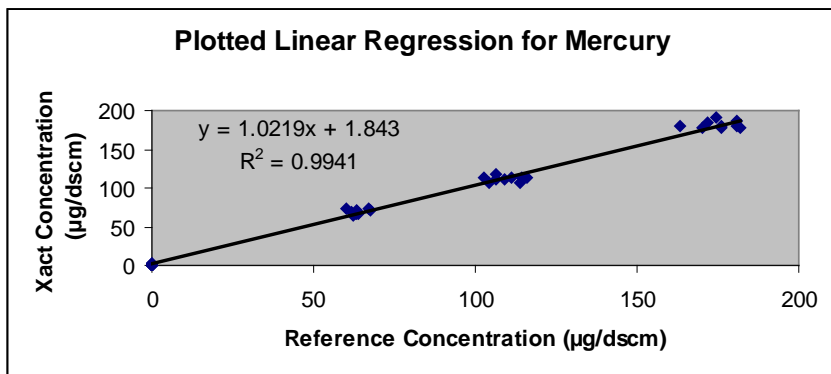


Figure 5. Plot of Linear Regression for Mercury from Xact CEMs

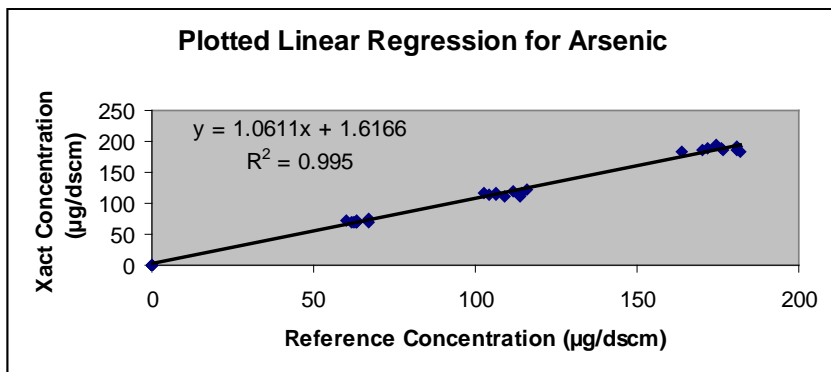


Figure 6. Plot of Linear Regression for Arsenic from Xact CEMs

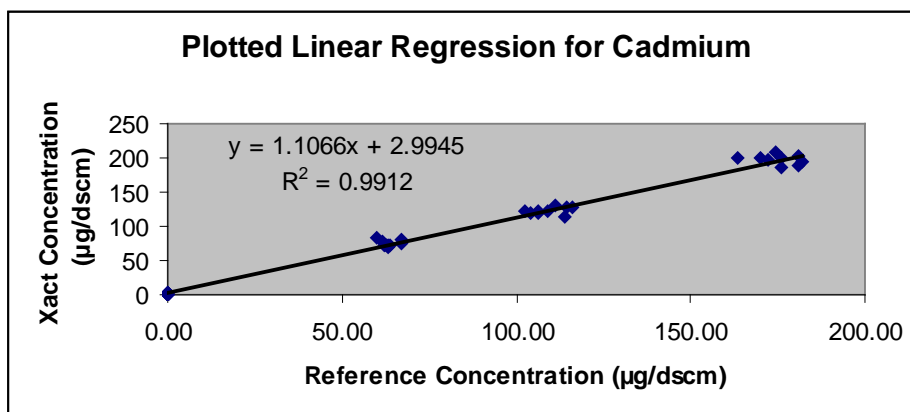


Figure 7. Plot of Linear Regression for Cadmium from Xact CEMs

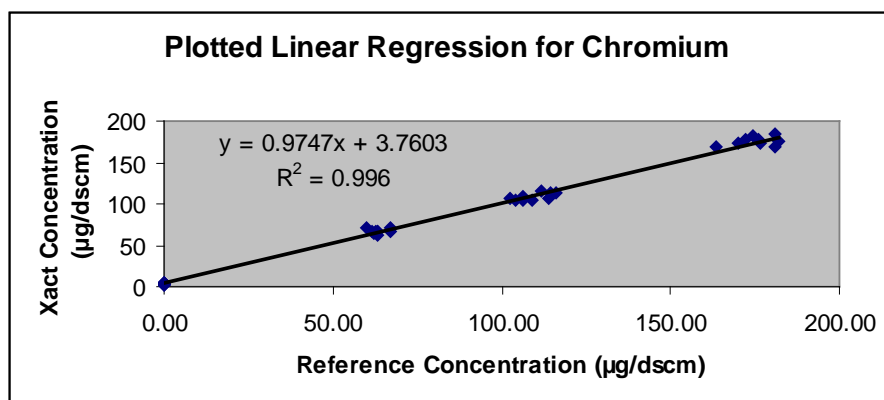


Figure 8. Plot of Linear Regression for Chromium from Xact CEMs

## COMPREHENSIVE PERFORMANCE TEST RESULTS

The T149 CPT was conducted September 28-29, 2010, utilizing the of a particulate matter CEMS, a multi-metals CEMS, and an HCl CEMS.

**Table 3. T149 HWC MACT Compliance Performance and Emissions Summary**

Parameter	Units	HWC MACT Standard	T149 2010 CPT Results			
			Run 1	Run 2	Run 3	Average
Stack gas particulate matter (Note a)	gr/dscf	0.013	0.013	0.012	0.012	0.012
Stack gas HCl/Cl <sub>2</sub> (Notes a, b)	ppmv, dry	32	4.2	3.0	3.9	3.7
Stack gas LVM (Note a)	µg/dscm	92	< 2.7	< 2.7	< 3.3	< 2.9
Stack gas SVM (Note a)	µg/dscm	230	19	17	18	18
Stack gas mercury (Note a)	µg/dscm	130	21	23	18	21

Notes:

(a) Corrected to 7% oxygen.

(b) HCl and Cl<sub>2</sub> combined, expressed as HCl or Cl<sup>-</sup> equivalents.

**Table 4. T149 CPT Particulate, HCl, and Arsenic Emissions Summary**

Parameter	Units	Run 1	Run 2	Run 3	Average
<b>Stack Sampling Parameters</b>					
Stack gas flow rate	dscfm	18,378	18,279	18,452	18,370
	dscm/min	520.5	517.7	522.6	520.2
Stack gas oxygen content	vol %, dry	9.0	9.0	9.4	9.1
<b>Particulate Emissions by Sigrist CTNR CEMS</b>					
Particulate concentration	gr/dscf	0.0127	0.0120	0.0125	0.0124
	gr/dscf @ 7% O <sub>2</sub>	0.0129	0.0115	0.0116	0.0120
	mg/dscm	29.1	27.4	28.5	28.3
	mg/dscm @ 7% O <sub>2</sub>	29.5	26.4	26.6	27.5
Particulate emission rate	lb/hr	2.00	1.88	1.97	1.95
	g/s	0.252	0.237	0.248	0.246
<b>Chloride Emissions by EcoChem MC3 CEMS</b>					
Stack gas HCl concentration as HCl or Cl <sup>-</sup>	ppmv, dry	2.76	1.69	2.44	2.30
	ppmv, dry @7% O <sub>2</sub>	4.23	2.98	3.93	3.71
Stack gas HCl emission rate as HCl	lb/hr	0.284	0.173	0.252	0.236
	g/s	0.0358	0.0217	0.0317	0.0297
Stack gas HCl emission rate as Cl <sup>-</sup>	lb/hr	0.276	0.168	0.245	0.230
	g/s	0.0348	0.0211	0.0308	0.0289
<b>Arsenic by Cooper Environmental Services XACT CEMS</b>					
Metal concentration	ug/dscm	0.95	0.88	1.04	0.96
	ug/dscm @ 7% O <sub>2</sub>	1.11	1.02	1.26	1.13
Metal emission rate	lb/hr	6.5E-05	6.0E-05	7.2E-05	6.6E-05
	g/s	8.2E-06	7.6E-06	9.1E-06	8.3E-06

**Table 5. T149 CPT Cadmium, Chromium, Lead and Mercury Metals Emissions Summary**

Parameter	Units	Run 1	Run 2	Run 3	Average
<b>Cadmium by Cooper Environmental Services XACT CEMS</b>					
Metal concentration	ug/dscm	2.40	2.57	2.52	2.49
	ug/dscm @ 7% O <sub>2</sub>	2.80	3.00	3.04	2.95
Metal emission rate	lb/hr	1.7E-04	1.8E-04	1.7E-04	1.7E-04
	g/s	2.1E-05	2.2E-05	2.2E-05	2.2E-05
<b>Chromium by Cooper Environmental Services XACT CEMS</b>					
Metal concentration	ug/dscm	1.26	1.36	1.63	1.42
	ug/dscm @ 7% O <sub>2</sub>	1.48	1.59	1.97	1.68
Metal emission rate	lb/hr	8.6E-05	9.3E-05	1.1E-04	9.7E-05
	g/s	1.1E-05	1.2E-05	1.4E-05	1.2E-05
<b>Lead by Cooper Environmental Services XACT CEMS</b>					
Metal concentration	ug/dscm	13.82	12.23	12.77	12.94
	ug/dscm @ 7% O <sub>2</sub>	16.10	14.33	15.45	15.29
Metal emission rate	lb/hr	9.5E-04	8.4E-04	8.8E-04	8.9E-04
	g/s	1.2E-04	1.1E-04	1.1E-04	1.1E-04
<b>Mercury by Cooper Environmental Services XACT CEMS</b>					
Metal concentration	ug/dscm	18.38	19.69	14.80	17.62
	ug/dscm @ 7% O <sub>2</sub>	21.42	23.04	17.82	20.76
Metal emission rate	lb/hr	1.3E-03	1.3E-03	1.0E-03	1.2E-03
	g/s	1.6E-04	1.7E-04	1.3E-04	1.5E-04

Note: dscf = Dry standard cubic feet

dscfm = Dry standard cubic feet per minute

Standard conditions are 68°F, 29.92 in. Hg (20°C, 760 mm Hg)

The following figures (Figures 9 -13) show the operating data from the T149 CEMs systems during the performance of the CPT.

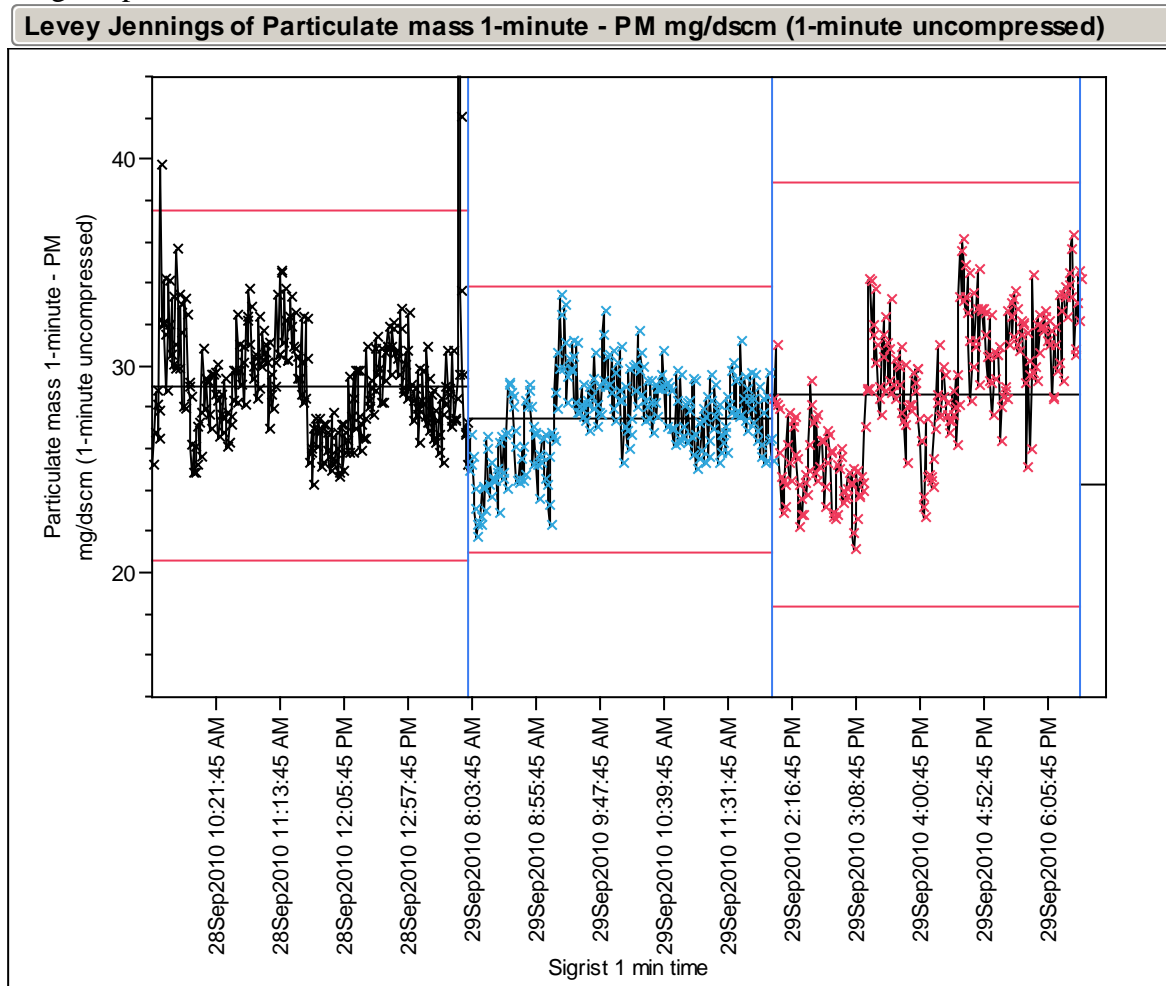


Figure 9.

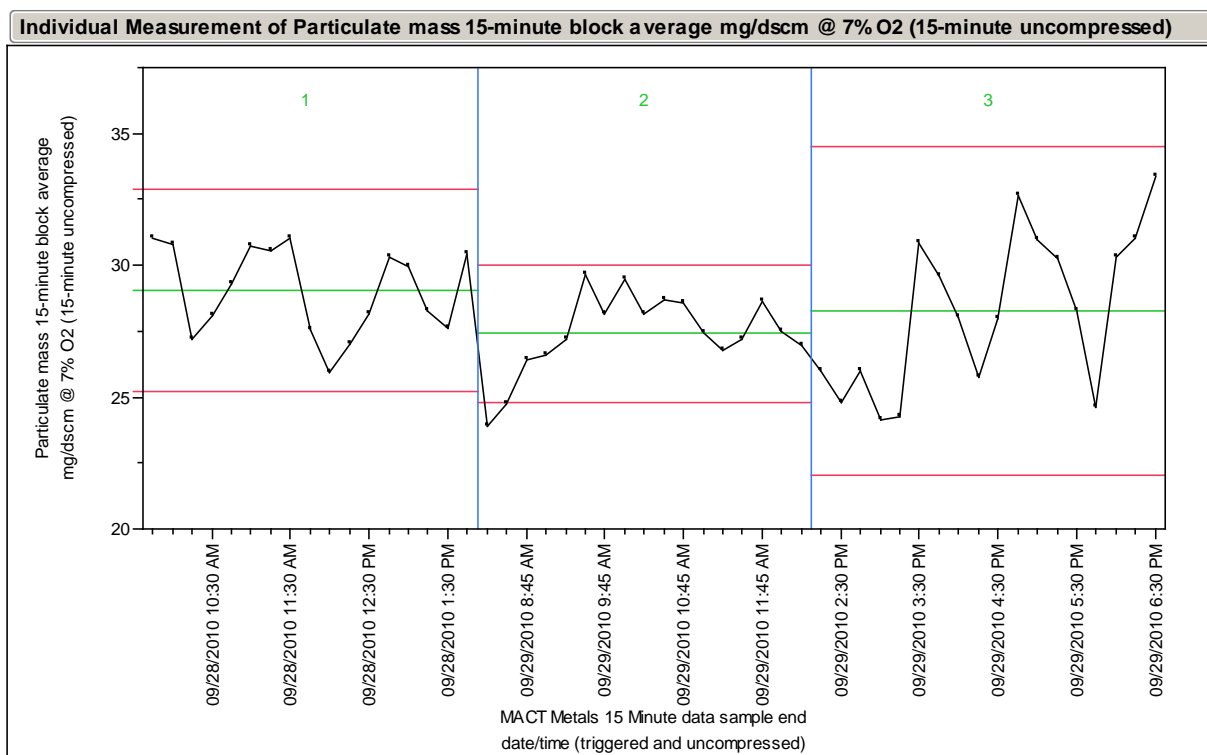


Figure 10.

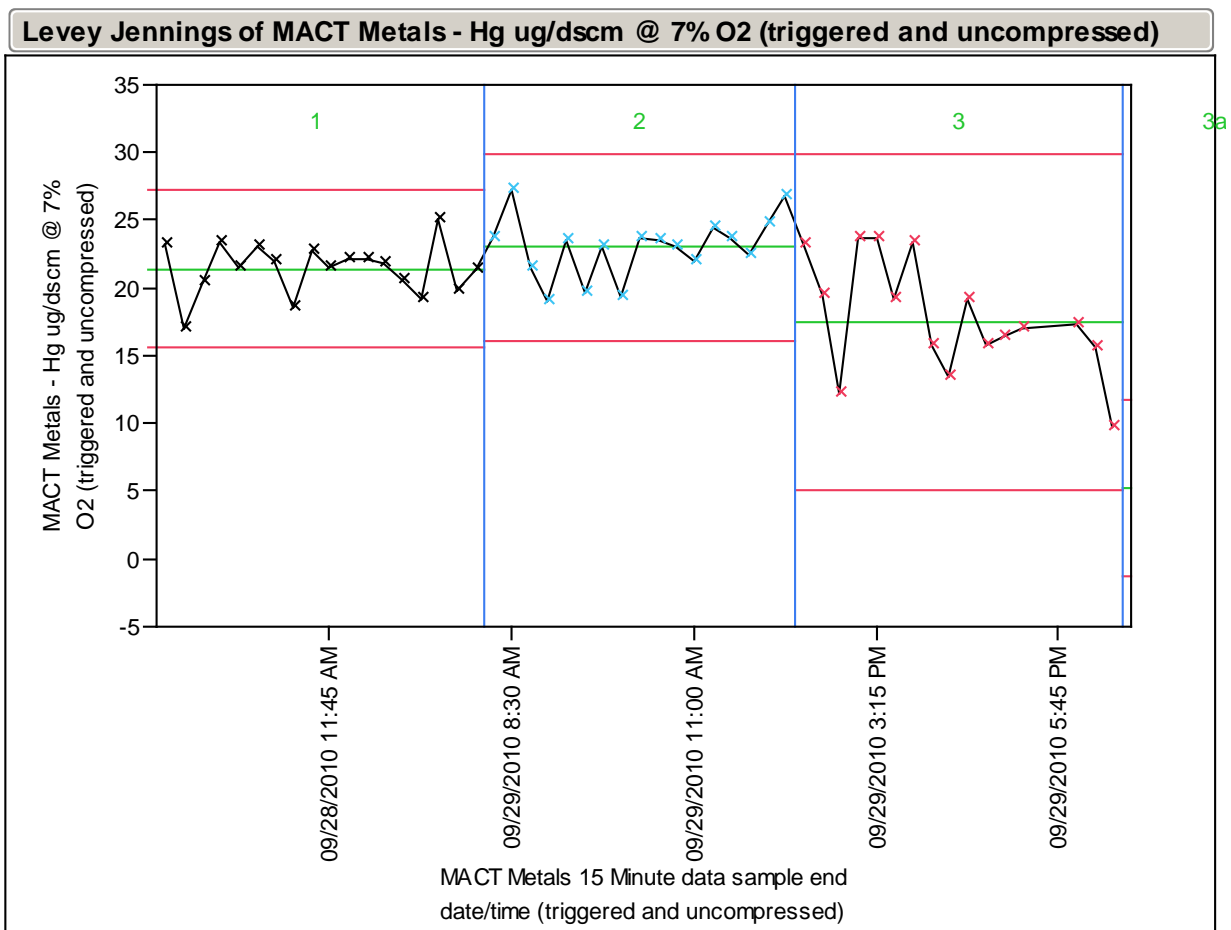


Figure 11.



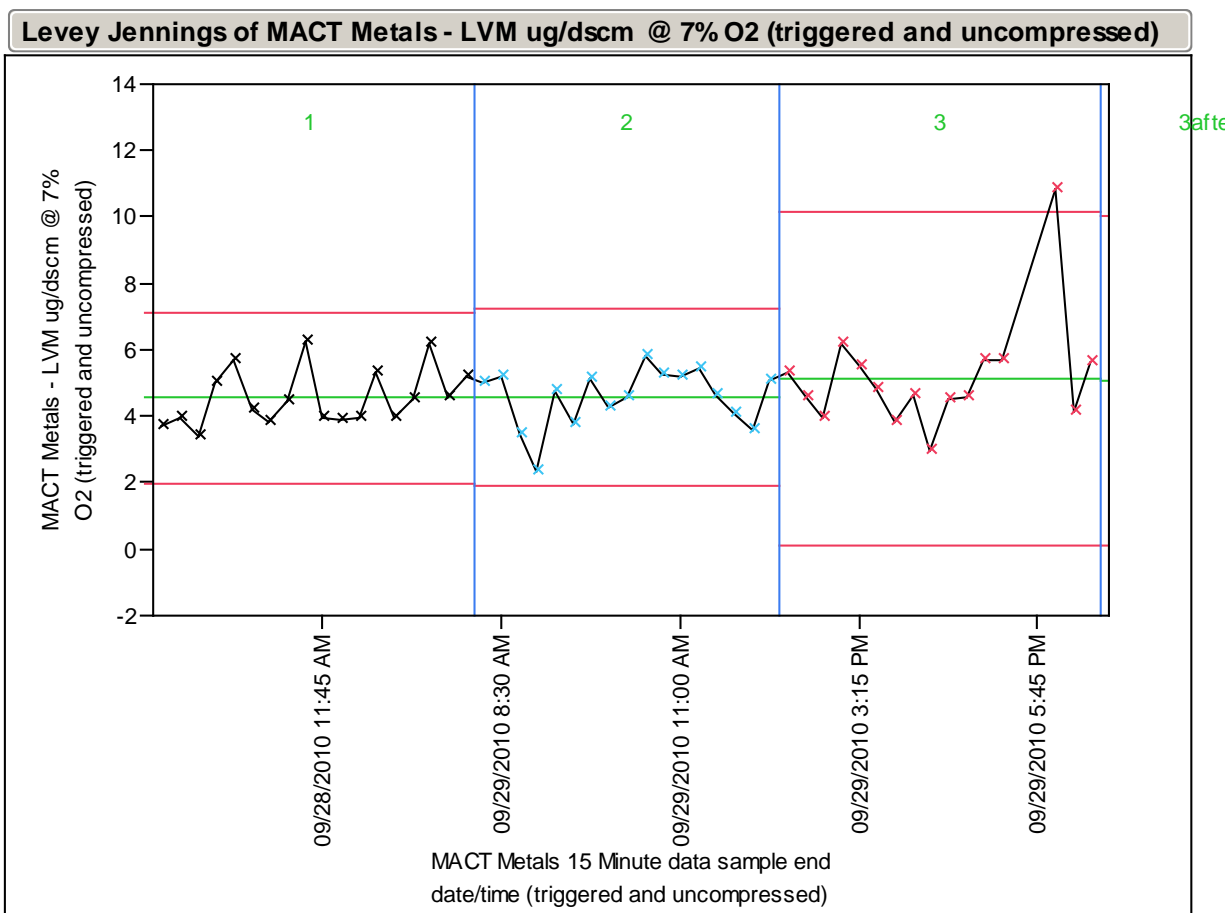


Figure 12.

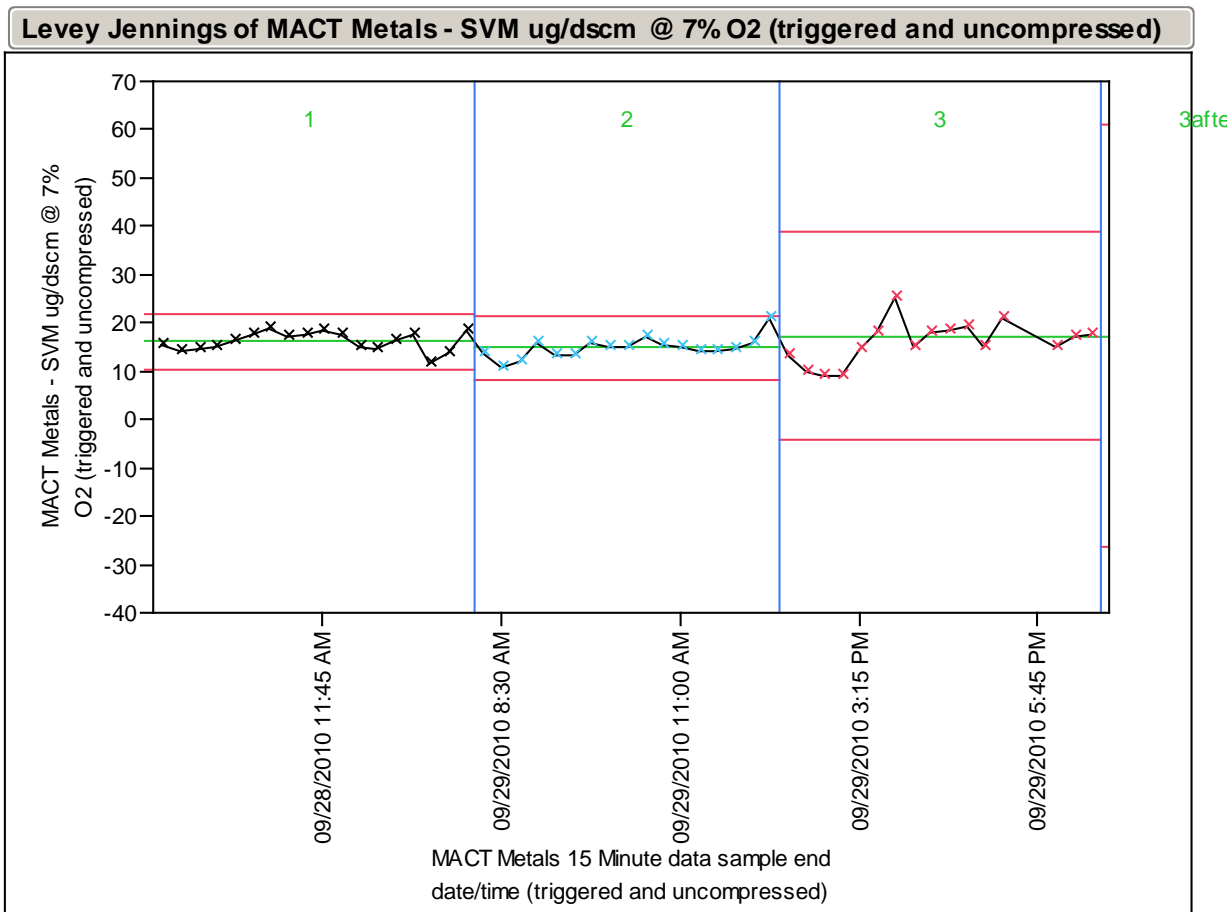


Figure 13.

Table 6. Metals SRE Comparison

Metal	2010 (CEMs)	2005 (Method 29)
Total LVM	99.998%	99.998%
Pumpable LVM	99.93%	99.85%
SVM	99.83%	99.74%
Hg	45.70%	17.6%

## **SUMMARY**

Specific conclusions drawn from the 2010 CPT are as follows:

- PM emission standard was met. A maximum ash feed rate limit can be appropriately developed from the T149 CPT results.
- Metal emission standards were met for Hg, SVM, and LVM. Maximum metal feed rates can be reliably and appropriately determined using the T149 CPT results.
- Stack gas HCl/Cl<sub>2</sub> emission standard was met. A maximum total chlorine feed rate limit can be appropriately established from the T149 CPT results.

Use of CEMs is a viable approach for CPT compliance demonstration.

For certain incineration facility, a number of benefits can be realized through use of CEMs for demonstration of compliance for emissions:

- Improved safety, improved operation efficiency, and economic advantages for waste characterization
- Operational flexibility for the incineration system
- Optimization of the establishment of operating parameter limits during the performance of the CPT.

## **ACKNOWLEDGEMENTS**

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## **KEY WORDS**

1. Continuous Emission Monitoring Systems
2. Hazardous Waste Combustor MACT Emission Compliance
3. Incinerator Emission Monitoring
4. Multi-Metals Continuous Emission Monitoring
5. Total Particulate Continuous Emission Monitoring
6. Hydrogen Chloride Continuous Emission Monitoring
7. Comprehensive Performance Test